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THE MICROMETALLURGY OF ACTINIUM

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ABSTRACT

Actinium metal has been prepared on the milligram scale by the reduction of actinium fluoride with lithium vapor. A high-purity product with a yield of 95% was obtained. It resembles lanthanum metal both chemically and physically. The melting point of actinium metal was found to be 1,050° ± 50°C. The relationship of the melting point of actinium metal to the melting points of metals adjacent to it in the periodic table is discussed.

INTRODUCTION

The chemical and physical properties of actinium metal and its compounds have not been exhaustively investigated, because actinium occurs in such small quantities in nature. Whereas radium occurs in uranium minerals to the extent of 1 part in 3,000,000 (parts of uranium), actinium occurs to the extent of only 1 part in 15,000,000,000. However, since the advent of nuclear reactors, it has been possible to obtain actinium in sizable quantities by neutron bombardment of radium-226 according to the reactions:[1]

$$Ra^{226}\ (n,\gamma)\ Ra^{227} \rightarrow Ac^{227} + \beta$$

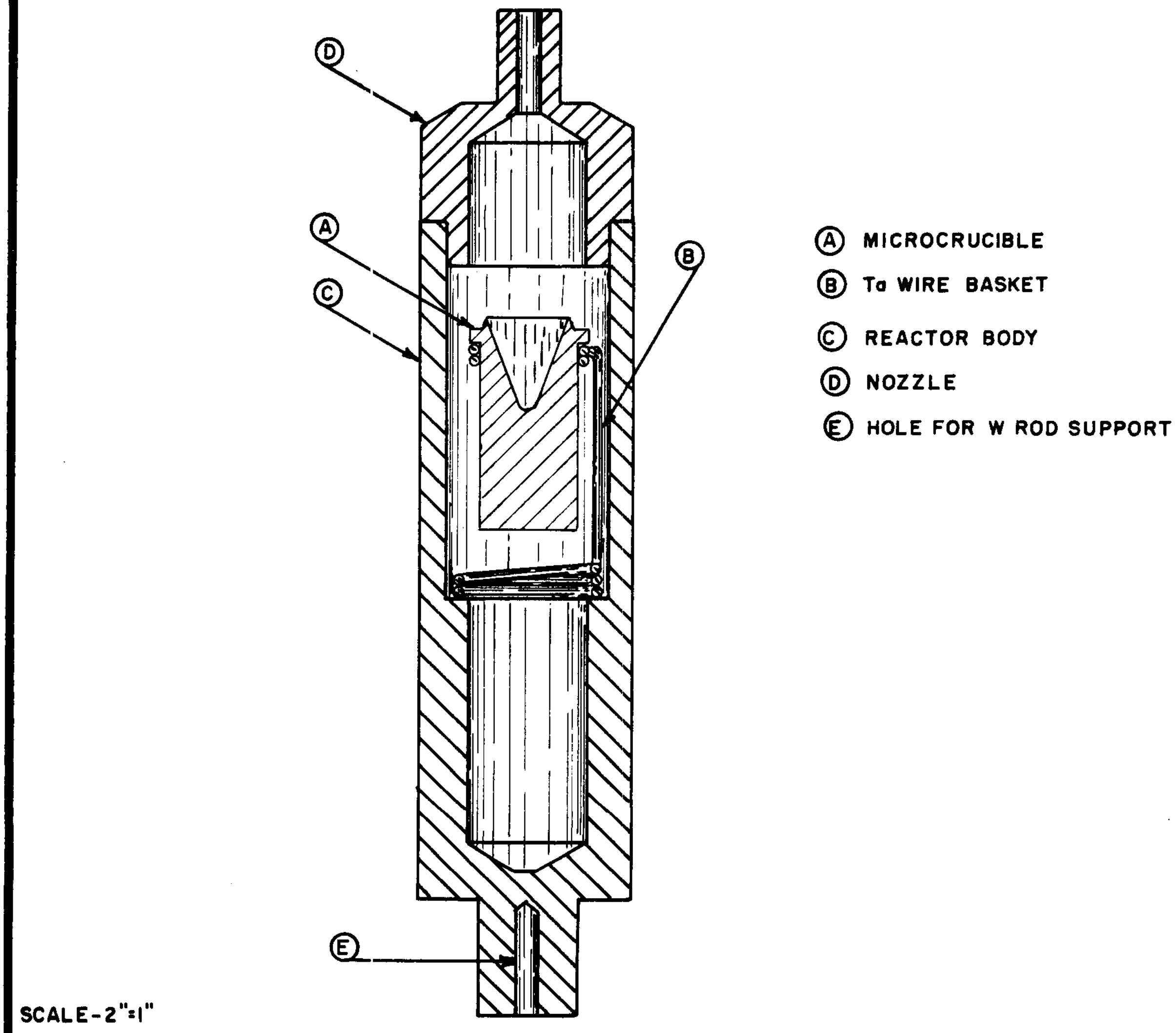
The actinium can then be separated chemically from the radium by the method of Hagemann.[2] Fried, Hagemann, and Zachariasen[3] studied the preparation and properties of some actinium compounds. These compounds were prepared in microgram quantities and were characterized by their X-ray diffraction patterns. The preparation of metallic actinium was not reported. As a continuation of this earlier work, actinium metal was prepared on a milligram scale in this laboratory, and a study of some of its chemical and physical properties was made. The metal was prepared by the lithium reduction of actinium fluoride in vacuum. The handling of actinium and its extraction from radium require the use of personnel shielding; therefore, most of the developmental work was carried out with lanthanum and its compounds as stand-ins for actinium.

EXPERIMENTAL

Apparatus

The lanthanum or actinium fluoride was contained in a molybdenum microcrucible, the inside dimensions of which were 0.200-inch diameter by 0.200-inch deep. This microcrucible was supported by means of a tantalum wire basket in a molybdenum reactor which consisted of a cylindrical body ($1\frac{13}{16}$-in. long by $\frac{9}{16}$-in. o.d.). The microcrucible and reactor assembly is shown in Figure 1. Two concentric holes were drilled into the reactor body - the first was 0.435-inch in diameter and 1-inch deep; the second hole, with a $\frac{5}{16}$-inch diameter and concentric with the first, extended $\frac{3}{4}$-inch deeper. The shoulder formed by the concentric holes served as a support for the wire basket. The lower hole served as a container for lithium metal pellets. The reactor body was capped by a nozzle which had a hole in the top, $\frac{1}{16}$-inch in diameter. The nozzle constricted the opening of the reactor to increase the pressure of the lithium vapor during a reduction. The nozzle and body of the reactor were wired together with tantalum wire to prevent the nozzle from being blown off.

The reactor was so supported in the vacuum envelope - by means of a cold finger tipped with a tungsten rod - that approximately the top one inch of the reactor was inserted into the coils of an induction heater. The lower part of the reactor was heated by conduction and thus was maintained at a somewhat lower temperature than the upper portion, which contained the microcrucible.



MICROCRUCIBLE AND REACTOR ASSEMBLY

FIGURE 1

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The length of the reactor was determined experimentally to give the desired rate of lithium distillation while still maintaining the upper portion at a temperature sufficiently high to initiate the reaction.

The vacuum envelope is shown in Figure 2. It is a double-walled Pyrex envelope capable of withstanding a vacuum of at least 5×10^{-5} millimeter of mercury. The double walls permit cooling of the envelope to prevent reaction of the glass with the reducing metal. The leads from the electronic heater were inserted into the envelope through side arms. A $\frac{65}{40}$ standard spherical male joint at the bottom of the envelope held the cold finger, which in turn supported the reactor. A similar joint at the top held a washing head used for flushing the system with methyl alcohol (to remove volatilized lithium) or with helium. The top surface of the washing head is optically flat, which permits the observation of the reactor during operations. The side arms, placed 180° from each other, are tapered downward to permit drainage of solvents used for washing and are terminated with $1\frac{1}{4}$-inch Housekeeper seals through which pass $\frac{3}{16}$-inch tubular copper leads for the induction coil. These copper leads are sealed with silver solder to the Housekeeper seals. The induction coil is $\frac{3}{4}$-inch i.d. by $1\frac{3}{8}$-inches long and consists of $5\frac{1}{2}$ turns of copper tubing. In operation, cooling water was passed through this coil. It was necessary to have the induction coils inside the vacuum envelope, since, for reproducible temperatures, the molybdenum reactor had to be pretreated at temperatures above 1,400°C. These temperatures were not attainable without close coupling.

The cold finger consists of two concentric Pyrex tubes and a $\frac{65}{40}$ standard spherical female joint. The outer tube is terminated with a $\frac{3}{4}$-inch Housekeeper seal onto which has been silver-soldered a $\frac{1}{2}$-inch by 0.080-inch tungsten rod which serves as a support for the reactor. The seal was made between the joints of the cold finger and the vacuum envelope by means of Apiezon N vacuum grease. Since this grease is quite viscous at room temperature, it was necessary to heat the female joint by means of a small resistance heater to facilitate the breaking of the seal by the rotating rod-runner.

The power for the induction coil was supplied by a five-kilowatt General Electric electronic heater. The six-step transformer on the heater was replaced by a 220-volt, 30-ampere Powerstat variable transformer which afforded a means of continuously varying the input to the high-voltage transformer.

The vacuum envelope was connected to the vacuum system through a $\frac{28}{15}$ standard spherical female joint which projects from one of the side arms. The vacuum was obtained through the use of a D.P.I. three-stage oil diffusion pump in conjunction with a Welch Duo-Seal or Cenco Megavac fore pump. The vacuum manifold was designed so that the envelope could be evacuated by the fore pump alone or by both pumps in series. It was also possible to open the envelope to atmospheric pressure without breaking vacuum on the pumps. A liquid-air cold trap was also incorporated in the vacuum manifold. The system could be evacuated to about 2×10^{-6} millimeter of mercury pressure, although the usual operating pressure is about 5×10^{-5} millimeter of mercury pressure. The pressure of the system was measured by means of a National Research Corporation thermocouple gauge and an ion gauge developed at this laboratory.[4]

Most of the work on actinium, except the actual loading of the microcrucible into the reactor, was carried out behind $\frac{1}{2}$-inch of lead shielding. The apparatus was observed through a $6\frac{1}{4}$ by $7\frac{1}{2}$ by 16-inch Lucite box filled with a saturated zinc bromide solution and through a 8 by 4 by 2-inch lead glass window. Mirrors were so located that a view of the top of the vacuum envelope could be obtained, thus facilitating the aligning of the cold finger and reactor within the envelope.

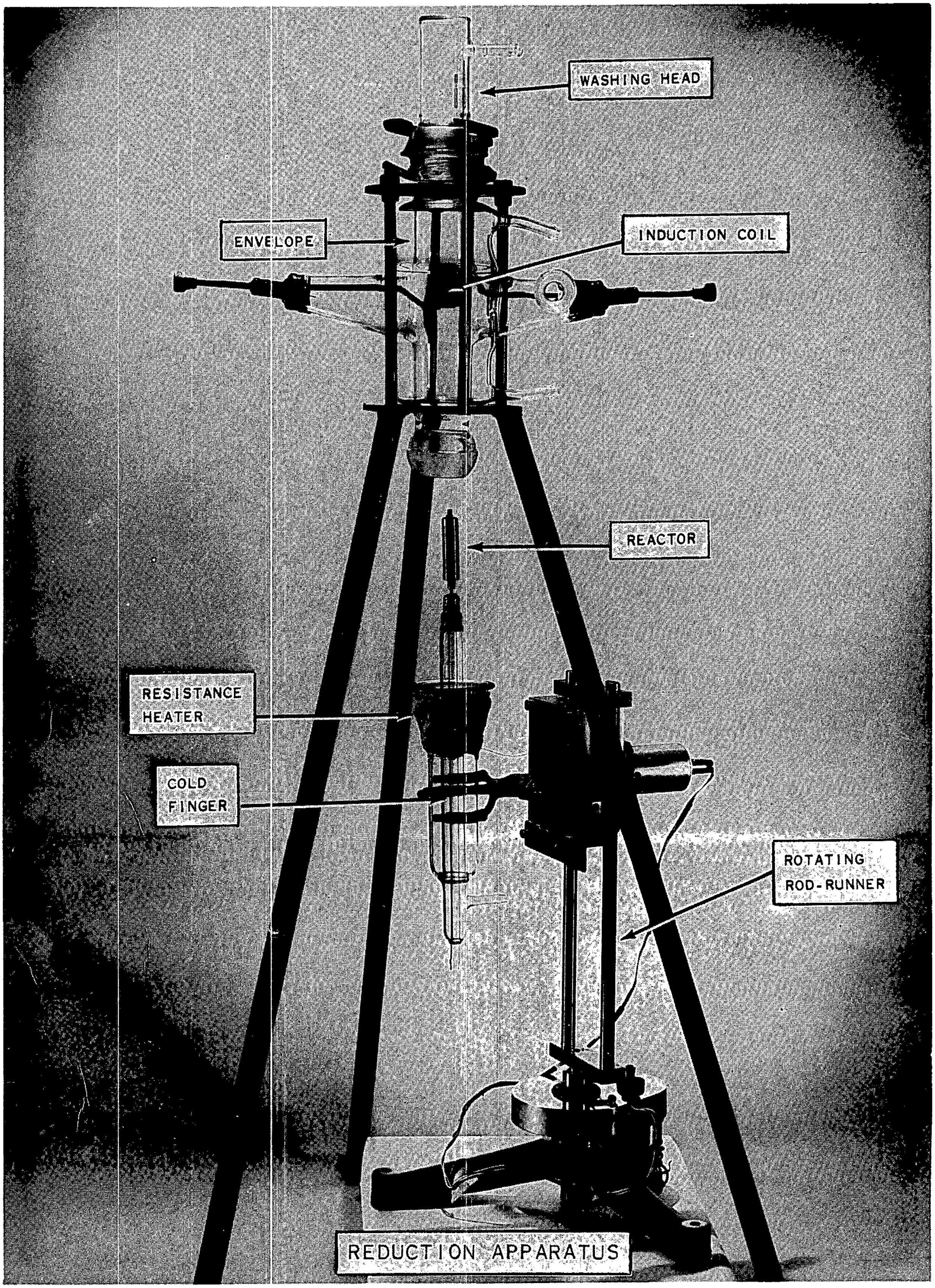
WASHING HEAD
ENVELOPE
INDUCTION COIL
REACTOR
RESISTANCE HEATER
COLD FINGER
ROTATING ROD-RUNNER
REDUCTION APPARATUS

FIGURE 2

The cold finger was inserted into and removed from the vacuum envelope by means of a 30-volt, direct-current, rotating rod-runner (Fig. 2). The rod-runner also served to move the cold finger into a helium-filled drybox used for loading and unloading the reactor.

Since lanthanum and actinium metals are both quite active chemically, they were handled at all times in a dry helium atmosphere. For weighing purposes, the microcrucibles were sealed in a helium-filled aluminum capsule (Fig. 3). The weighing capsule is made of anodized aluminum and consists of three parts: the base, inner cover, and outer screw-cover. The microcrucible containing the activity fits into the hole in the base. An "O" ring seals the inner cover to the base. The inner cover is held in place by pressure applied by the outer screw-cover. The "O" ring seal proved satisfactory for maintaining a helium atmosphere within the capsule.

The capsule is protected from dust, etc., by a small bell jar which fits over a Lucite base in which the capsule sits. The assembly and disassembly are accomplished through the use of Lucite tools. (It was necessary to fabricate tools of a soft material to prevent abrasion of the capsule.) With repeated use, it is possible to obtain reproducible weights to ±0.02 milligram, if sterile techniques are employed.

A Fisher Semi-micro Gramatic Balance, Model No. 1-911, was used for the weighings. This balance weighs to ±0.02 milligram with constant sensitivity, has a capacity of 100 grams, and is operable behind barriers. Approximately 30 to 45 seconds is required for a weighing.

Standardization of Heater

The efficiency of the reduction reaction is dependent upon the temperature; thus, it is required that there be some means of temperature determination and control. A Leeds and Northrup Model 8622-C optical pyrometer was used for temperature determination. The observed temperatures were corrected for the absorption of the zinc bromide window and for the emissivity of molybdenum.[5] The observed temperature was that of the exterior of the reactor and thus somewhat higher than that of the interior. Since it was impossible to observe the reactor during a reduction because of the formation of the lithium coat on the envelope, it was necessary to calibrate the temperature of the reactor against the output of the induction heater. The output was measured by means of a Ballentine electronic voltmeter, the antenna for which was wrapped around the lower part of the cold finger. A calibration curve of temperature *versus* Ballentine voltmeter reading has to be made for each set of equipment, since the temperature is dependent upon the nature and dimensions of the leads, the coils, and the reactors, and upon the pressure of the system. It was impossible to obtain an accurate comparison of temperature *versus* Powerstat setting, since the output is dependent upon line voltage, which is not constant.

Since the heating of a substance in an induction field is a surface phenomenon, the attainment of reproducible temperatures is possible only if the nature of the surface is reproducible. Molybdenum slowly reacts with the oxygen in air to form a superficial coat of the oxide. It was found that reproducible temperatures could be obtained only if this superficial oxide coating was previously removed by volatilization at >1,400°C and at 5×10^{-5} millimeter of mercury pressure.

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fig 3

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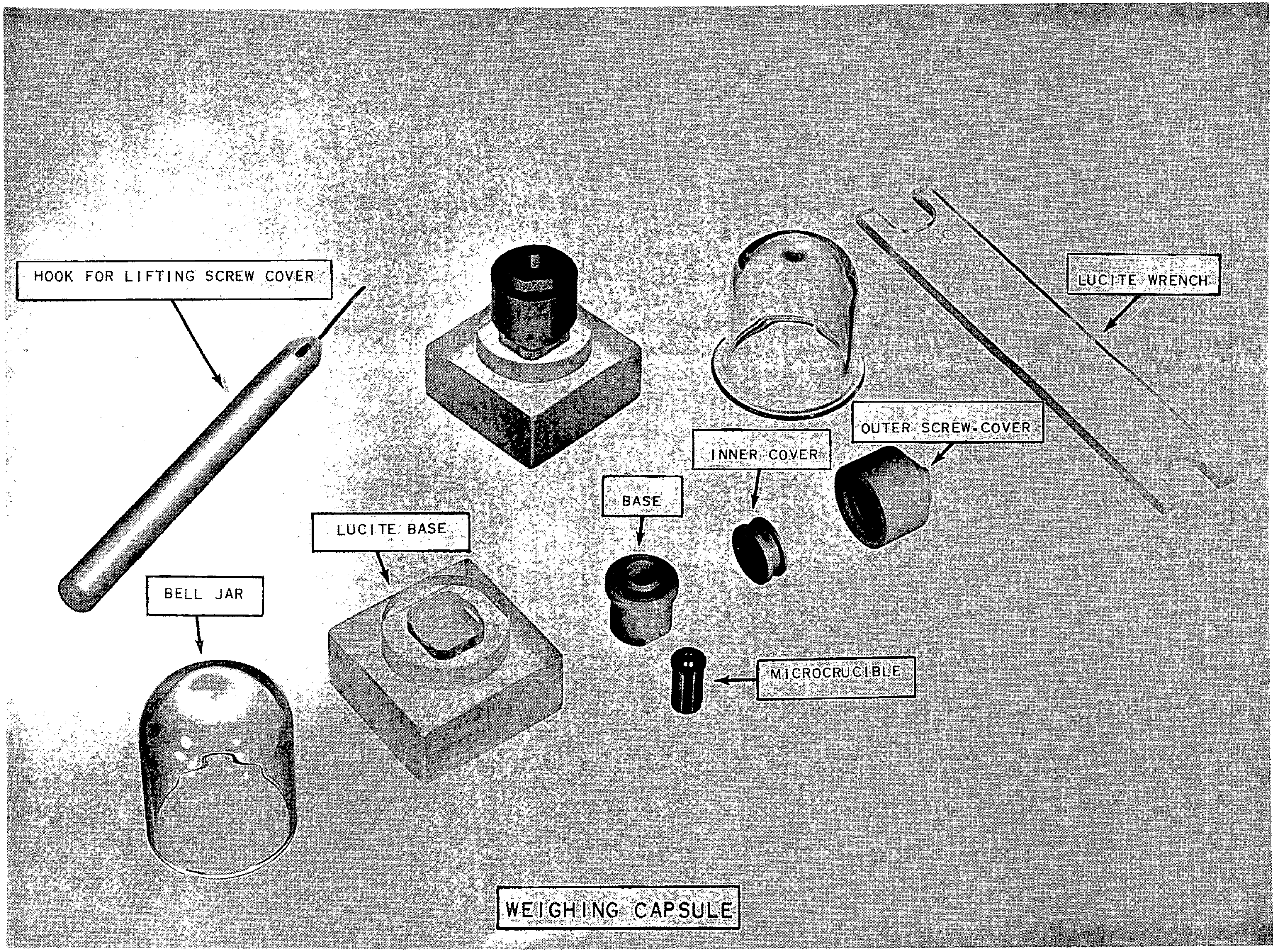

FIGURE 3

Reagents

LITHIUM

Lithium metal rods of $\frac{3}{8}$-inch diameter, packed in an inert atmosphere, were obtained from Metalloy Corporation. These rods were cut in an atmosphere of helium into small pellets which would fit easily into the lower section of the reactor.

ACTINIUM AND LANTHANUM FLUORIDES

The same procedure was used for the preparation of both fluorides. The fluorides were prepared in a Teflon centrifuge tube[6] which was constructed in such a way that a molybdenum microcrucible formed the bottom of the tube. The microcrucible was held in place by means of a screw which pressed it against the bottom of the Teflon cone.

The fluorides were precipitated by adding an aliquot of the metal chloride solution to approximately five milliliters of 24 per cent hydrofluoric acid solution in the centrifuge tube. About five milliliters more of the acid was added, and the mixture was stirred. Then the mixture was centrifuged for 15 minutes at 1,800 revolutions per minute, in a Size 1, Type SB International Clinical Centrifuge. The fluoride formed a gelatinous precipitate in the microcrucible. The precipitate was dried for about 30 minutes with an infrared lamp and then for $1\frac{1}{2}$ hours in an oven at 200°C. The anhydrous fluoride formed a hard, dense pellet which did not adhere to the crucible. Yields of 99.7 per cent and purities of >99.4 per cent of theoretical were consistently obtained by use of this procedure.

The actinium was extracted from irradiated radium by a technique similar to that used by Hagemann.[2] However, when actinium fluoride was precipitated from the product solution, it contained a large percentage of iron, aluminum, and magnesium, which interfered with the reduction reaction to the extent of making it impossible. Therefore, the actinium as obtained by the Hagemann process was further purified by a double acid oxalate precipitation and ignition to the oxide, which was then dissolved in hydrochloric acid. Actinium fluoride precipitated from this solution was completely reduced.

Procedure

The microcrucible containing the fluoride pellet was placed into the reactor, which had been loaded with 150 to 200 milligrams of lithium metal pellets. The nozzle was wired in place on the reactor, and the assembly was placed on the cold finger. The reactor was then inserted into the vacuum envelope and aligned in the coils to prevent a short circuit. The system was evacuated to 5×10^{-5} millimeter of mercury pressure. Then for 25 minutes, the reactor was heated inductively to about 1,000°C in the case of lanthanum and to 1,200°C in the case of actinium.

At the end of the 25-minute heating cycle, the reactor was cooled for five minutes in vacuum and then for five additional minutes in an atmosphere of helium. The microcrucible was then removed from the apparatus and weighed. The purity of the lanthanum samples was determined spectrographically. The neutron growth rate of actinium was followed as a means of detecting light-element impurities.[7]

DISCUSSION

Rare earth halides are usually used as starting material for the production of rare earth metals.[8] To reduce rare earth (or, more specifically, lanthanum) halides, a strong reducing agent is required. Thus, a survey of alkali and alkaline earth metals and their halides was made from the standpoint of both their chemical and physical properties. The thermodynamics of the possible reactions between lanthanum halides and active metals was studied to determine which combinations would be most feasible.

The vapor pressures of the various metals and halides were obtained from Brewer's tables.[9] From examination of the vapor pressures, it is noted that at higher temperatures the vapor pressures of both lithium bromide and lithium iodide are above that of lithium metal. In the reaction:

$$LaX_3 + 3M \rightarrow 3MX + La$$

such an observation is significant, since the halide of the reducing metal would be removed as the reducing metal was distilled, i.e., if lanthanum bromide or lanthanum iodide could be reduced by lithium vapor, the equilibrium would be shifted to favor the formation of lanthanum metal by volatilizing the lithium halides. On the other hand, the reduction of lanthanum fluoride by calcium or barium would not be desirable, since calcium fluoride and barium fluoride have vapor pressures approximately the same as that of lanthanum fluoride (or actinium fluoride).

The tables of thermodynamic and physical properties of the metals and metallic halides collected by Brewer and co-workers[9] permit the calculation of equilibrium constants for a number of reduction processes. These constants are useful in a qualitative way in determining which processes might be feasible; however, it must be remembered that many of the values given in these tables are necessarily only estimates, although probably the best estimates available. Further, it must be remembered that in these processes the thermodynamic equilibria may be greatly influenced by other factors, such as relative volatilities of the reactants and products, which determine the yield of a given process to a large extent.

The tables cited give values of the modified free energy function

$$\bar{G} = \frac{\Delta F - \Delta H_{298}}{T} \qquad (1)$$

for the various metallic halides, in which ΔF is the standard free energy of formation at absolute temperature (T), and ΔH_{298} is the heat of formation at 298°K. Since

$$\Delta F = \Delta H_{298} + \bar{G}T \qquad (2)$$

the standard free energy change in reduction processes (ΔF_t) may be calculated from these standard free energies for the formation of the compounds concerned. Thus, for the reduction

$$RX_3 + \frac{3}{n}M \rightarrow R + \frac{3}{n}MX_n$$

$$\Delta F_t = \frac{3}{n}\Delta F_{MX_n} - \Delta F_{RX_3} \qquad (3)$$

where RX_3 is an appropriate trivalent metallic halide and M an active metal reducing agent of valence n. If Equation (2) be substituted into Equation (3), then

$$\Delta F_t = \frac{3}{n}(\Delta H_{298} + \bar{G}T)_{MX_n} - (\Delta H_{298} + \bar{G}T)_{RX_3} \qquad (4)$$

then, by rearrangement

$$\Delta F_t = T(\frac{3}{n}\bar{G}_{MX_n} - \bar{G}_{RX_3}) + (\frac{3}{n}\Delta H_{MX_n} - \Delta H_{RX_3})_{298} \qquad (5)$$

Since

$$\Delta F_t = -RT \ln K \tag{6}$$

then, equating (5) and (6)

$$\ln K = \frac{1}{R}(\overline{G}_{RX_3} - \frac{3}{n}\overline{G}_{MX_n}) + \frac{1}{RT}(\Delta H_{RX_3} - \Delta H_{MX_n})_{298} \tag{7}$$

In Table I are collected the calculated equilibrium constants (K) at 1,000°, 1,250°, and 1,500°K for a number of possible reduction reactions. Large values of K indicate that a given reaction is thermodynamically favorable. Thus, it is evident that the sodium or potassium reduction of lanthanum or actinium fluoride would not be favorable.

TABLE I

EQUILIBRIUM CONSTANTS

REDUCING METAL	HALIDE	1,000°K	1,250°K	1,500°K
		LANTHANUM		
Li	F	1.0×10^{3}	1.3×10^{2}	2.7×10^{1}
	Cl	8.0×10^{6}	2.4×10^{5}	1.9×10^{4}
	Br	6.9×10^{6}	6.0×10^{5}	1.4×10^{5}
	I	8.1×10^{7}	9.1×10^{6}	2.6×10^{6}
Na	F	6.6×10^{-6}	5.0×10^{-5}	2.4×10^{-4}
	Cl	2.2×10^{4}	1.7×10^{3}	3.8×10^{2}
	Br	4.6×10^{5}	2.5×10^{4}	4.6×10^{3}
	I	4.5×10^{7}	1.9×10^{6}	3.2×10^{5}
K	F	5.3×10^{-8}	9.8×10^{-7}	1.0×10^{-5}
	Cl	6.6×10^{7}	7.6×10^{5}	4.5×10^{4}
	Br	1.5×10^{10}	6.9×10^{7}	2.3×10^{6}
	I	3.7×10^{12}	5.9×10^{9}	1.0×10^{8}
		ACTINIUM		
Li	F	1.7×10^{3}	2.4×10^{2}	2.7×10^{1}
	Cl	1.7×10^{8}	3.3×10^{6}	2.0×10^{5}
	Br	3.2×10^{7}	2.2×10^{6}	4.6×10^{5}
	I	1.1×10^{7}	2.7×10^{6}	1.6×10^{6}
Na	F	1.1×10^{-5}	9.5×10^{-5}	5.5×10^{-4}
	Cl	4.6×10^{5}	2.3×10^{4}	4.0×10^{3}
	Br	2.1×10^{6}	8.9×10^{4}	1.5×10^{4}
	I	5.9×10^{6}	5.6×10^{5}	2.0×10^{5}
K	F	8.7×10^{-8}	1.9×10^{-6}	2.4×10^{-5}
	Cl	1.4×10^{9}	1.0×10^{7}	4.8×10^{5}
	Br	6.9×10^{10}	2.6×10^{8}	7.6×10^{6}
	I	4.9×10^{11}	1.7×10^{9}	6.0×10^{7}

The calcium or barium reduction of lanthanum and actinium fluorides, chlorides, or bromides is not desirable, since the vapor pressures of the calcium and barium halides are about equal to or less than those of the corresponding lanthanum or actinium halides.[9] Calcium might be used for the reduction of the iodides, since the vapor pressure of calcium iodide is greater than that of lanthanum or actinium iodide.

Thus, it seems that (1) the most feasible reducing agents to study would be the alkali metals, i.e., lithium, sodium, and potassium - probably increasing slightly in usefulness in that order; (2) anhydrous lanthanum or actinium iodide, bromide, chloride, and fluoride are all suitable for reduction, the completeness and probable ease of reduction decreasing in that order, as judged from consideration of equilibrium constants and relative volatilities; (3) calculated equilibrium constants alone indicate a decreasing yield with rising temperature; however, in general the volatility of the product metal halide increases more rapidly than that of the metal, which increases the yield as the temperature rises. The latter effect is probably greater, so temperatures of 1,000°C or higher should favor the reduction.

The stability and ease of preparation of the anhydrous lanthanum (and actinium) halides increase in order from the iodide to the fluoride. The anhydrous iodides are quite difficult to prepare,[8,10] while the anhydrous fluoride may be prepared by precipitating from water solution and drying the precipitate.[8,11]

The fluorides can be reduced to the metal by lithium, calcium, or barium; however, lithium is the most satisfactory, since its fluoride is the most volatile. Therefore, it was concluded that in view of the ease of preparation of lanthanum and actinium fluorides, the ease and comparative safety of handling lithium metal, and the feasibility of the reaction, the lithium reduction of lanthanum and actinium fluorides would be investigated. The problem in the first stages was thus resolved to the reduction of milligram quantities of anhydrous lanthanum fluoride by lithium vapor at about 1,000°C and at a pressure sufficiently low to volatilize the lithium fluoride formed in the reaction. As a result of this developmental work with lanthanum, actinium metal was prepared.

RESULTS

Actinium metal was obtained as small metallic pellets adhering to the microcrucible bottoms. Its physical appearance resembled that of lanthanum metal. The pellets were usually a silvery-white color, although occasionally they had a golden cast. The actinium metal glowed weakly in the dark with a characteristic blue color. Actinium metal, like lanthanum metal, is a very active metal chemically. The metal pellet oxidized rapidly in moist air to form a coat of white actinium oxide which was somewhat effective in preventing further oxidation.

Data for some typical reductions of both lanthanum and actinium fluorides are shown in Table II. An average yield of 98.4 per cent was obtained for the lanthanum reductions. Spectrographic analyses showed that these metal preparations contained less than 0.1 per cent fluorine.[12]

In the preparation of actinium metal, an average yield of 94.5 per cent was obtained. The percentage of fluorine was calculated from neutron counts on the assumption that all the neutrons were due to the fluorine.[7] All of the metal preparations contained less than 0.3 per cent fluorine on this basis.

TABLE II

YIELD AND PURITY OF METAL PREPARATIONS

LANTHANUM

WEIGHT LaF_3 (mg)	CALC (mg)	FOUND (mg)	YIELD (%)	FLUORIDE (%)
11.43	8.10	8.00	98.8	<0.1
11.45	8.12	7.99	98.4	<0.1
13.45	9.56	9.48	99.2	<0.1
22.83	16.19	15.29	94.4	<0.1
24.89	17.65	17.77	100.6	<0.1
25.98	18.43	18.19	98.7	<0.1

ACTINIUM

WEIGHT AcF_3 (mg)	CALC (mg)	FOUND (mg)	YIELD (%)	NEUTRONS PER SECOND*	CALC MAX FLUORIDE (%)
6.23	4.98	4.62	92.8	5,000	0.30
8.64	6.90	6.30	91.3	5,000	0.22
12.30	9.83	9.76	99.3	2,000	0.06

*COUNTED AT EQUILIBRIUM

The temperature of reduction is critical. If too low a temperature is used, the product metal does not melt and incomplete reduction results; if too high a temperature is used, some of the halide is volatilized and a low yield results. For lanthanum, reductions carried out at 950°C were incomplete and reductions carried out above 1,100°C resulted in low yields. For actinium, the temperature range for maximum yield and purity was 1,100° to 1,275°C.

The experiments indicate that the melting point of actinium metal is about 200°C higher than that of lanthanum. The reported melting points of lanthanum metal are 826°C[13] and 887°C[9]. The apparent melting point of actinium metal is 1,050° ± 50°C.

CONCLUSIONS

The relationship of the melting point of actinium to that of adjacent metals is shown in Table III, which is a partial periodic table of melting points.

The melting points which were obtained from Brewer's tables[9] for the elements in the first four A-subgroups of Periods 3, 4, 5, and 6 are listed. For any given period, the melting points increase from Group I to IV. In agreement with this observation, the melting point of actinium falls between that of radium and thorium. In general, the melting points of the elements in the various groups decrease with increasing atomic number. However, this observation does not appear to be valid for Period 6. The reported melting point of radium (960°C) is higher than that of barium (717°C). We observed a similar anomaly for actinium (1,050°C) and lanthanum (887°C). Since the melting points of the metals are related to their atomic volumes,[14] the anomalies are probably due to the effect of lanthanide contraction. The elements affected by lanthanide contraction are those in Table III below the heavy line. In Group IV, the effect of lanthanide contraction is observed in the fifth period. The melting point of hafnium (2,327°C) is higher than that of zirconium (1,860°C). Therefore, the melting point for actinium shows both a horizontal and vertical relationship to the melting points of its neighboring elements.

TABLE III

PARTIAL PERIODIC TABLE OF MELTING POINTS*

	GROUP							
	I		II		III		IV	
PERIOD								
3	K	64	Ca	851	Sc	1,397	Ti	1,727
4	Rb	39	Sr	771	Y	1,477	Zr	1,860
5	Cs	29	Ba	717	La	887	Hf	2,327
6	Fr	?	Ra	960	Ac	1,050	Th	1,827

*ALL TEMPERATURES ARE REPORTED IN °C.

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